FCS09 – SOP for Operating and Maintaining GC-FID Instruments

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1. Scope

1.1. This document establishes the procedures for preventative maintenance and quality control that apply to instrumental analysis. The purpose of these maintenance and quality control (QC) procedures is to ensure that instruments are working properly and are free of contaminants before processing casework.

2. Background

2.1. To establish a procedure for regular instrument maintenance and QC to ensure quality and accuracy of reported casework results.

3. Safety

3.1. Reagent Toxicity: Personnel should refer to the appropriate SDS for solvents and reagents used during analysis for any specific safety requirements.

3.1.1. For a complete review of required Health and Safety regulations of the PHL, see *DOM13 – DFS Health and Safety Manual*.

3.2. Protective Equipment: Personnel should wear personal protective equipment (PPE) including: lab coat, gloves, and safety goggles when carrying out standard operating procedures.

3.2.1. Wear vinyl or nitrile gloves when handling these chemicals to prevent absorption through the skin. If any chemicals are spilled onto gloves, discard gloves into hazardous waste.

3.3. Training: Formal training in use of instruments and software is necessary.

3.4. Personal Hygiene: Universal Precautions must be followed. Care should be taken when handling chemicals or any biological specimen. Routine use of gloves and proper hand washing should be practiced.

3.4.1. Refer to *DOM13 – DFS Health and Safety Manual*.

3.5. Disposal of Waste: Waste materials must be disposed of in compliance with laboratory, Federal, state, and local regulations. Solvents and reagents should always be disposed of in an appropriate container clearly marked for waste products and temporarily stored in a chemical fume hood.

3.5.1. Consult DFS Safety Officer for proper procedures.

4. Materials Required

4.1. Reagent Grade Acetonitrile (ACN) and Methanol (MeOH), Chloroform ($CHCl_3$), or higher purity.

4.2. Gas Chromatograph-Flame Ionization Detector (GC-FID)

4.2.1. Fully assembled, including: solvent vials, injection syringe, and other consumables.

4.2.2. Ultra-High Purity Helium tanks

4.2.3. Hydrogen Generator

4.2.4. Computer monitor equipped with instrumental software and attached printer.

4.3. Binder/Folder for Standard results, or electronic equivalent

4.4. Maintenance Logbook(s)

5. Standards and Controls

5.1. Certified drug standard (Ex: Cocaine), or equivalent positive control

6. Calibration

6.1. Not applicable

7. Procedures

7.1. Daily Maintenance Procedure

7.1.1. Check the helium levels in the setup in the hallway.

7.1.2. Update gas signage as appropriate.

7.1.3. Replace helium tanks as appropriate.

7.1.4. Check hydrogen generator water level. Add water when necessary.

7.2. Weekly Maintenance Procedure

7.2.1. Weekly Maintenance must be carried out each week on each instrument used for casework and must be performed prior to any other casework. If only one instrument is used for casework, only the instrument being used must undergo this weekly maintenance.

7.2.2. Each Monday is considered the start of a new week for weekly maintenance purposes (or the following business day if Monday is a holiday).

7.2.3. Solvent Vials

7.2.3.1. Empty the waste vials in the autoinjector tray.

7.2.3.2. Empty and replace Acetonitrile and/or Methanol in the solvent vials in the autoinjector tray.

7.2.3.3. Empty and replace Acetonitrile and/or Methanol in the solvent blank vials in the autoinjector tray.

7.2.4. Syringe Cleaning

7.2.4.1. Clean autoinjector syringe using Acetonitrile and/or Methanol.

7.2.5. Sequence

7.2.5.1. Save Sequence from the previous week.

7.2.5.2. File the previous week's sequence in the GC-FID Logbook, or electronic equivalent.

7.2.5.3. Load the Weekly Sequence template file and save as a new sequence using the current date with the format YYYY.MM.DD (Ex: 2015.12.25).

7.2.6. Positive and Negative Controls

7.2.6.1. Run a negative control (blank run) in the SCRN165.M method to ensure that there are no contaminants present in the system.

7.2.6.2. If contaminants are present, additional blanks shall be run until a clean blank is obtained.

7.2.6.3. If a clean blank cannot be obtained, the machine shall be taken out of service until the cause of contamination is found and removed.

7.2.6.4. Run a positive control (Standard run) to ensure the system is functioning properly.

7.2.6.5. The positive control must clearly display the peaks of interest with no significant background.

7.2.6.6. Record if the blank is clear and whether or not, it shows all expected peaks for standard.

7.2.6.7. Record the retention time of each peak.

7.2.6.8. File the standard run and the blank before it in the GC-FID Logbook.

7.2.7. Preventative Maintenance Schedule

7.2.7.1. Change Liner: Monthly/as needed.

7.2.7.2. Replace water in hydrogen generator as needed.

7.2.7.3. Cut/Replace Column: as needed.

7.2.7.4. Replace Ultra-high Purity Helium tanks, as needed.

7.3. Quality Control Procedure

7.3.1. A negative control (blank run) shall be run prior to each sample using the BLANK method or the same method used in analysis. The blank must be free of discernible peaks in order to be acceptable.

7.3.2. Sample run printouts shall be accompanied by the previous blank's printout in order to ensure the system was free of contaminants prior to the run of interest.

7.3.3. Each sample and standard vial must be labeled with unique identifying marks to ensure that the correct sample or standard is run.

7.3.4. When a run is complete, the vial number listed on the printouts should be compared to the corresponding vial in the autosampler tray to ensure that the correct sample is run.

7.3.5. **Acceptance Parameters** – The following are the acceptance parameters for the GC-FID.

	Acceptance Criteria	Detail
GC-FID PARAMETERS	**Retention Time**	Retention time of analyte peak must match within 2% of standard or within 0.05 minutes, whichever is higher.
	S/N* Cut Off	Analyte signal intensity must be more than three (3) times greater than noise.
	Peak width resolution	Analyte peak must be base peak resolved, as evaluated by the analyst.

*S/N = Signal-to-Noise Ratio

7.4. Sample and Standard Run Procedure

7.4.1. In order to identify a substance in a sample, a pure standard corresponding to the analyte of interest must be run during the same week as the sample and must be listed on the same sequence file as the sample. The substance must be run on the same method as the standard. For confirmation, this method shall be used in conjunction with a SWGDRUG Category A test, ASTM E2329-17, of which results shall be in agreement.

7.4.2. A separate standard shall be run for each analyte of interest being reported, but multiple samples may be matched up with the same standard.

7.4.3. A blank run must be performed before the standard as if it were a sample.

7.4.4. The retention time of the sample must be within 2% (or within 0.05 minutes, whichever is higher) of the retention time of the standard in order to be declared a match. Because concentrations of analytes can affect their retention times, if the retention time is outside this range, the sample or standard may be diluted with corresponding solvent and rerun.

7.4.5. A copy of the matching standard printout and the blank run immediately before it shall be included with the printout of the sample.

7.4.6. A copy of the printout for each standard used to match casework and the blank run immediately before it shall be retained in the Standards Logbook.

7.5. Control Chart Maintenance

7.5.1. As appropriate, the significant parameters appropriate for the identification of individual substances or mixtures of heroin, cocaine, etc. (or other substance used for quality control) shall be recorded in the laboratory control chart for GC-FID. Critical pieces of information include peak retention time.

8. Sampling

8.1. See *FCS02 – SOP for General Laboratory Procedures for FCU*

9. Calculations

9.1. Not applicable

10. Uncertainty of Measurement

10.1. See *FCS21 – Procedure for Uncertainty in Measurement*.

11. Limitations

11.1. Not applicable

12. Documentation

12.1. Maintenance Logbooks and Control Charts

13. References

13.1. DFS Departmental Operations Manuals, (current revisions).

13.2. Forensic Chemistry Unit Quality Assurance Manual, (current revisions)

13.3. Forensic Chemistry Unit SOPs, (current revisions).

13.4. ASTM E2329-17, Standard Practice for Identification of Seized Drugs.